RiO5 METHOD (23)

OCG-MEL-XMU

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^{226}Ra and ^{228}Ra—Manganese-oxide Fiber leaching—Seawater sample

Leaching Mn-fiber for ^{226}Ra and ^{228}Ra for Gamma counting

Table of Contents

1 SCOPE

This method specifies the minimum requirements and laboratory methods for measuring ^{226}Ra and ^{228}Ra in seawater samples using gamma-spectrometry via leaching Mn-fiber.

Water samples are collected in acid-cleaned containers and pretreated on board, by passing through a 1 mm cartridge filter followed by a MnO_2-impregnated acrylic fiber (Mn-fiber) column to extract dissolved radium (see Rama and Moore, 1996). Post processing will take place at the home laboratory by leaching the Mn-fiber and co-precipitating radium with $BaSO_4$. Ra isotopes emissions are finally measured by gamma spectrometry (see Moore, 1984). Isotope ingrowth and decay corrections are applied to calculate ^{226}Ra and ^{228}Ra activities.

2 EQUIPMENT CHEMICAL REAGENTS

2.1 Equipment

- Plastic containers
- Standard laboratory equipment
- Glass beakers
- Hot plate with magnetic stirrer
- Analytical balance with an accuracy of ± 0.1 mg
- Filter
- Gamma spectrometry system
- Centrifuge

2.2 Tracers

- No tracers

2.3 Chemical reagents

- Hydroxylamine hydrochloride (HAHCl)
- Hydrochloric acid (HCl)
- Barium nitrate ($Ba(NO_3)_2$)
- Sodium bisulfate ($NaHSO_4$)

2.4 Solutions

- 1 M HAHCl

  Add 70 g HAHCl to a 1 litre measuring cylinder and add Milli-Q water to make up to 1 L
- 1 M HCl
- Saturated $Ba(NO_3)_2$

- 1 m $NaHSO_4$

3 PROCEDURE

1. Collect water samples and pass the samples through a 1 mm cartridge filter followed by a MnO_2-impregnated acrylic fiber (Mn-fiber) column to extract dissolved radium.

The extraction step usually takes place in situ and the following steps are done at the home laboratory.

2. Prepare 1 M solutions of hydroxylamine hydrochloride and 1 M HCl. Store these stock solutions separately.

3. Mix 2 volumes of HAHCl with 1 volume HCl for the number of samples to be leached.

For example to leach 8 samples, mix 1500 ml HAHCl with 750 ml HCl. This mixture may be stored if you mix too much. Label it Mn-Fiber leach Solution: 2 HAHCl + 1 HCl.

4. For each Mn-fiber sample, put the fiber in a 1000 ml beaker and add enough of the leach solution to cover the fiber (250-300 ml) and heat to 80-90°C (no boiling). Stir the fiber occasionally to expose all fiber to solution. If some black spots remain, add 5 ml leach solution each time until all fiber turns white.

5. Remove from heat and cool to room temperature.

6. Pour the solution and fiber into a filter cup and filter through a glass fiber filter using a suction flask and a new filter. Wring out fiber. Suck all the solution into the flask. Rinse fiber with 280 ml 1M HCl followed by 100 ml MilliQ water. Wring out fiber again. Combine all washings in a 1000 ml beaker.

7. Add 5 ml of saturated $Ba(NO_3)_2$ solution and mix well. Then add 25 ml 1 M $NaHSO_4$. Mix well to form $BaSO_4$ precipitate.

8. Let solution stand overnight. Decant most of supernatant liquid. Transfer the precipitate to a 100 ml beaker.

9. Let precipitate settle and use a pipette transfer it into a centrifuge tube used for Gamma counter.

10. Centrifuge and decant excess solution several times. Keep the supernatant liquid to 2 cm height in the tube. Cover the tube. Label with the sample name.

11. On a log sheet note the date the sample was precipitated. Samples must age 3 weeks after precipitation before counting by gamma spectrometry.

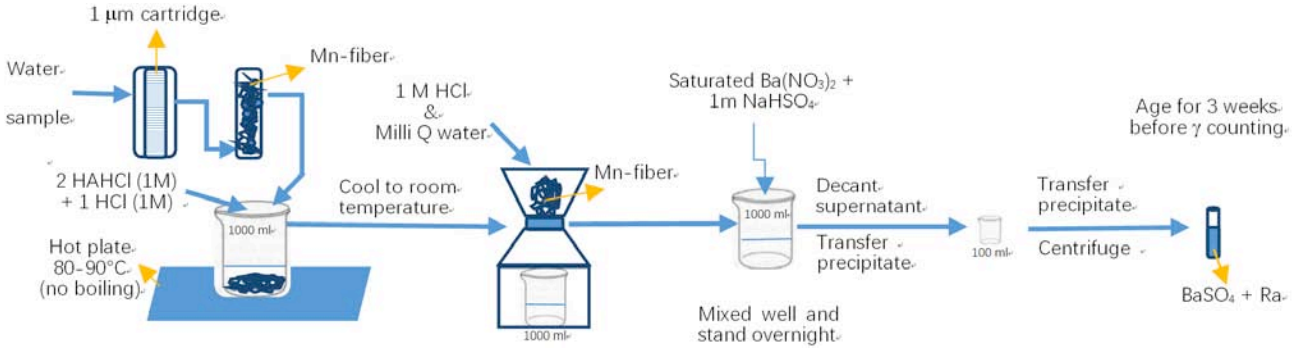
12. After counting, in-growth and decay corrections are then applied to calculate ^{226}Ra and ^{228}Ra activities at sampling time.

4 REFERENCES

Moore W. S. (1984) Radium isotope measurements using germanium detectors. *Nucl. Inst. Methods* **223**, 407–411.

Rama and Moore W. S. (1996) Using the radium quartet for evaluating groundwater input and water exchange in salt marshes. *Geochim. Cosmochim. Acta* **60 (23)**, 4645–4652.

5 FLOW CHART



6 IMAGES


Image 1. Ra in water samples extracted onto Mn-fibers.

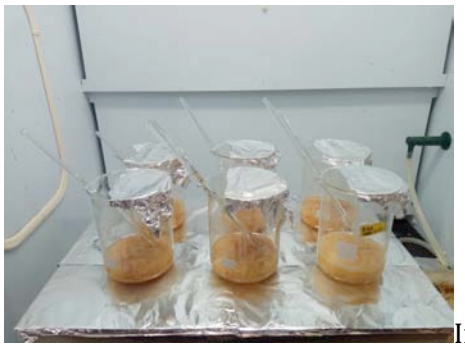


Image 2. Acid-leaching of Mn-fibers.


Image 3. Co-precipitation of Ra by $BaSO_4$.

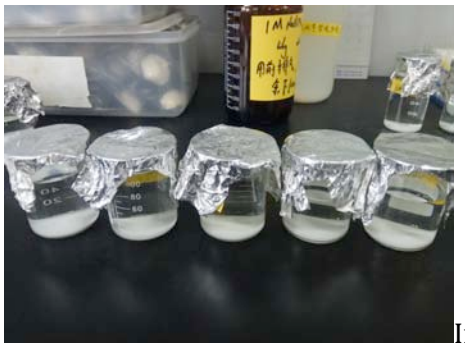


Image 4. After standing overnight the supernatant and precipitate.

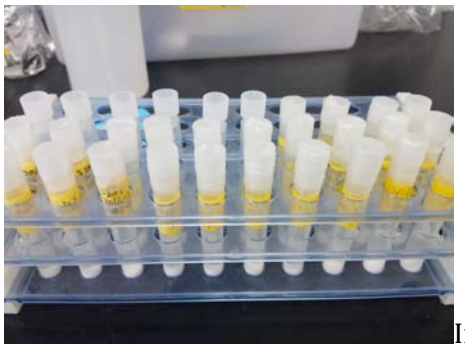


Image 5. Precipitate transferred into counting vials that will be counted after aging for 3 weeks.